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(54) Process for treating wet paper with foam.

(57) Specifically structured foamed compositions of paper  
treating agents applied to wet paper, such as wet-end paper  
web produced during high speed paper manufacture, pro-  
vide uniform treating agent distribution and treated paper  
having properties comparable to dry-processed paper.

EP 0 195 458 A1

PROCESS FOR TREATING WET PAPER WITH FOAM  
BACKGROUND OF THE INVENTION

Field of the Invention

5        This invention pertains to processes for treating wet paper, especially wet paper webs produced during paper manufacture, and more particularly to such processes using foamed compositions containing paper treating agents.

Description of Background Information

10       Paper manufacture, using what is known in the art as wet-laid papermaking, involves the production of non-woven paper sheets, known as paper webs, from raw materials, i.e., wood pulp, using an aqueous suspension of the paper fibers in water.

15       Papermaking using such wet method processing typically involves forming a pulp slurry consisting of up to a few weight percent of paper fiber suspended in water. The fibers are cast onto a screen, such as wire belts, where water is drained off to produce wet-end paper web. The web may also

20       pass through a press section to "squeeze out" more water. Such wet-end paper webs, either pressed or unpressed, are then dried, typically using air and/or heat, to reduce the moisture content in the paper down to generally less than ten weight percent providing dry-end paper product.

25       Paper treating agents, including performance or functional chemicals, are applied during the papermaking process. These agents can be added to assist the papermaking processing by adding

30       D-13,973

process performance chemicals or can be added to the paper as product performance chemicals.

Various procedures, such as squeeze rolls, sprays, slots or blades, have been used to apply paper treating agents during papermaking operations with limited success.

Squeeze rolls involve contacting the paper web with a composition containing treating agent, in either a liquid or foam, immediately prior to passing through the rolls. Limitations in the properties of treating agent, which can be applied by squeeze rolls arise due to various limitations in their use. The treating agent compositions must generally be low-viscosity, low-solids compositions since strong hydraulic forces generated in squeeze rolls at high speeds separate the rolls leading to loss of control in the amount of composition applied material and other problems. Studies show that fluid penetration mechanisms govern pick-up such that a strength gradient from the paper surface to the sheet center develops, unless saturation occurs. The significant increase in moisture content of the paper from squeeze roll applications requires added drying steps and apparatus significantly increasing cost and prevents their use in wet-end processing.

Spraying paper treating agent compositions onto paper web also has limited utility. Generally, only low-viscosity liquids can be utilized. Limitations in the uniformity of the application of treating agent exist. Sensitivity to spray nozzle design and performance, as well as inherent

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limitations in spray configurations and overlap, leads to inefficient distribution. Spraying often provides uneven deposition, particularly for wide paper webs which are typically used in papermaking.

5 Spraying is also sensitive to air currents which may affect uniformity of deposition. Certain treating agents may not be used for environmental reasons due to risk of being spread through the air.

10 These and other apparatus which apply liquid compositions, such as utilizing distribution slots or bars, knife blades or the like, require burdensome drying operations. Such liquid applications cannot be used on wet-end paper which cannot support the weight of the liquid applied to

15 the paper web. Furthermore, absorption of paper treating agents from liquid reservoirs followed by doctoring off the excess requires a paper web of uniform moisture content to enable uniform absorption.

20 Treatment of wet-end paper during papermaking operation is limited due to the inherent weakness in the paper web having high moisture content. Wet-end paper web has a weak structure which cannot absorb significant amounts of water without becoming unmanageable. As a consequence, 25 much paper treatment is directed toward dry-end processing. Due to significant wet pick-up, i.e. addition of water to the paper substrate, such processing is inapplicable to the wet-end processing.

30 The application of foamed treating compositions to dry-end paper webs is described in  
U.S. - A - 4,158,076. Uniform

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distribution of treating agent onto the paper web is disclosed using a foam application zone having an opening in, or upstream of, the area where foam contacts the paper web. It is disclosed from column 6, line 65 to column 7, line 21, that filling the application zone with foam precludes obtaining an acceptable deposition of treating agent, either through uneven and uncontrolled deposition or through a partial or total loss of deposition.

Foamed compositions have been applied to paper webs passing through the screening area of papermaking machines using suction to draw a foam onto the web, as disclosed in U.S. - A -

4,081,313                    The disclosed process is designed to apply the foamed composition to the paper web without touching it or compressing it in any way by direct mechanical contact.

A process for treating porous substrates with foamed treating compositions is described in DE-A-2,722,083. The application describes a large class of porous substrates which may be treated with foamed treating compositions including textile fabric or a non-woven material, paper, or wood veneer.                    Said application is directed primarily towards, and contains specific embodiments for, a process for producing a fabric treated

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uniformly and which is generally essentially dry to the touch, although it is disclosed that the porous substrate need not be dry. All the specific examples treat textile fabric as the porous substrate.

5 Textile materials are air permeable to a relatively high degree, which property facilitates the separation of air from liquid in a foam stream at contact with the fabric or textile. In contrast, 10 many paper materials such as unfinished writing papers, book papers, newsprint linerboard, boxboard, containerboard, and the like, are substantially non-porous being relatively low in permeability. Such papers are also, in comparison to textiles, 15 relatively low in absorbency and very slow in absorbency rate of liquids. The relatively low level of absorbency and particularly the low rate of liquid absorbency presents serious difficulties in obtaining suitable treating agent distributions from 20 foamed compositions at high rates of paper processing such as those used in commercial papermaking.

25 Foam applicators used in applying foamed treating compositions to a substrate such as a textile or fabric, are described in U.S. -A- 4,023,526.

Foamed compositions used in related treating processes are described in U.S.

- A - 4,099,913 .

30 It would be desirable to provide a process for uniformly distributing paper treating agents onto wet paper web during paper manufacture including under, normal wet-end conditions. Such a

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process must provide low wet pick-up due to the  
weakness of wet paper webs. The process should be  
applicable to a wide range of paper treating agents  
including high-solids, high viscosity compositions.  
5 The process should enable the uniform deposition of  
treating agent independent of paper web properties,  
such as moisture content. The process must also be  
applicable to relatively non-porous paper materials  
and operate effectively at high speed papermaking  
10 operations.

#### SUMMARY OF THE INVENTION

This invention pertains to a process for  
treating wet paper. This process comprises the  
following essential steps. A first step comprises  
15 (A) producing a foam containing gas and a liquid  
treating composition comprised of liquid vehicle,  
paper treating agent and foaming agent. The foam  
has a density of from about 0.005 to  
0.8 g/cm<sup>3</sup>, an average bubble size of from  
20 0.05 to 0.5 millimeters in diameter and  
a foam half-life of from 1 to 60  
minutes. A second step comprises (B) passing the  
foam through at least one foam applicator nozzle  
filling an orifice between upstream and downstream  
25 lips of the nozzle with the foam. A third step  
comprises (C) passing a wet paper web having a  
moisture content of at least about 5 weight percent  
across the nozzle in the direction of the upstream  
lip to the downstream lip. A fourth step comprises  
30 (D) applying a controlled amount of the foam to the  
surface of the wet paper web providing a uniform  
distribution of the paper treating agent onto the  
wet paper web.

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This invention further pertains to a process for producing treated paper. This process comprises the following essential steps. A first step comprises (A) producing a wet paper web having a moisture content of at least about 5 weight percent. A second step comprises (B) producing a foam containing gas and a liquid treating composition comprised a liquid vehicle, paper treating agent and foaming agent. The foam has a density of from 0.005 to 10 0.8 g/cm<sup>3</sup>, an average bubble size of from 0.05 to 0.5 millimeters in diameter and a foam half-life of from 1 to 60 minutes. A third step comprises (C) passing the foam through at least one foam applicator nozzle filling an orifice between upstream and downstream lips of the nozzle with the foam. A fourth step comprises (D) passing the wet paper web across the nozzle in the direction of the upstream lip to the downstream lip. A fifth step comprises (E) applying a controlled amount of foam to the surface of the wet paper web to provide a substantially uniform distribution of the paper treating agent onto the wet paper web. A sixth step comprises (F) drying the wet paper web. A seventh step comprises (G) recovering the dried, treated paper web.

#### DETAILED DESCRIPTION OF THE INVENTION

This invention provides an improved process which enables the uniform distribution of paper

treating agents onto wet paper, such as wet-end paper webs produced during papermaking operations. The improved process is applicable to a wide range of paper treating agents, including high solids, high viscosity compositions. The improved process is applicable to the manufacture of substantially non-porous paper web with a low rate of liquid absorbency and is effective under typical papermaking operations including high rates of paper processing. The improved process operates substantially independent of moisture content variations in the wet paper and provides low wet pick-up effective in treating wet-end paper containing moisture contents up to near saturation.

The class of paper materials treated by the process of this invention pertains to paper webs or substrates, such as those produced in wet-laid papermaking operations. The process of this invention is particularly applicable to substantially non-porous paper relatively low in permeability. Substantially non-porous paper has a relatively high fiber content. As such, non-porous paper has a relatively high density as compared to the density of the fiber per se. Depending upon the fiber weight and paper material, substantially non-porous paper generally has a density of over 30 weight percent the density of fiber in the paper. For example, natural wood fiber has a density of 1.55 g/cm<sup>3</sup> such that non-porous paper of such fiber will typically have a density of from about 0.5 to more than 1.0 g/cm<sup>3</sup>. Illustrative paper webs used in this invention include non-porous paper such as

unfinished writing papers, book papers, newsprint, linerboard, boxboard, containerboard and the like, as well as porous paper such as tissues, filtration grade paper and the like.

5           The paper webs used in the process of this invention may be produced by wet-laid papermaking machines following those procedures well established in the art. In a typical procedure, a pulp slurry, usually comprised of from       0.1 to       5  
10           weight percent fiber in water, is cast onto a moving wire belt or screen where water is drained to produce a wet-laid non-woven web of paper, usually having a moisture content of up to about 90 weight percent. The paper web may be passed through a  
15           multistage press section where more water is "squeezed out" to produce a paper web having a moisture content of up to about 75 weight percent.

20           The wet paper used in the process of this invention has a moisture content of from at least about 5 weight percent up to the saturation level of the paper web. Preferably, the wet paper has a moisture content characteristic of wet paper webs produced in wet-laid papermaking operations. The wet paper will preferably have a moisture content of  
25           from       5 weight percent to       40 weight percent, and most preferably from       10 weight percent to       30 weight percent.

30           Although the process of this invention is primarily directed toward the treatment of in-process, wet-end paper webs during papermaking operations, the process would be equally applicable to previously dried paper wetted to have a moisture content of at least about 5 weight percent.

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Liquid treating compositions used in the process of this invention contain the paper treating agent, a liquid vehicle and usually a foaming agent. The paper treating agent is the active material which is distributed onto the paper web by the process of this invention. The liquid vehicle is generally required as a carrier to assist in the deposition of the paper treating agent onto the paper web. The paper treating agent may be provided in the liquid vehicle in any form, such as by dispersion, emulsification, solvation, or other means known in the art.

The paper treating agent used in the process of this invention pertains to the class of materials recognized by those skilled in the art as having utility when applied to paper substrates. Typical paper treating agents include functional and performance chemical additives for paper, such as product performance and process performance chemicals. Illustrative paper treating agents include sizing aids such as starches, casein, animal glue, synthetic resins including polyvinyl alcohol and the like materials which may be applied to the pulped slurry or to the formed sheet; binders, including wet strength or dry strength resins, such as polymers and copolymers of acrylamide, acrylonitrile, polyamide, polyamine, polyester, styrene, ethylene, acrylic acid, acrylic esters and materials such as rosin, modified, gums, and glyoxal; coloring agents including dyes and pigments such as the class of direct, reactive and fluorescent dyes and pigments or whitening agents, such as titanium dioxide, or organic

color types commonly used to color paper; oil or water repellants; defoamers to the extent the foaming agent is not rendered inoperative; fillers; slimicides; latex; saturants; wax emulsions; and the like. Blends of more than one paper treating agent 5 may be used.

Preferred paper treating agents include: sizing aids such as starches; binders, including wet-strength or dry-strength resins such as 10 polyamides, phenolics, and acrylics; crosslinkers such as melamine formaldehyde resins; and coloring agents.

The concentrations of paper treating agent is not critical so long as an effective amount 15 is provided to the paper web to provide treated paper having the desired properties, based on well-established practices in the art. The particular concentration of paper treating agent desired will vary depending upon the particular type 20 of paper treating agent, rate of foam application, rate of moving paper, paper properties and the like considerations, which determine the amount of paper treating agent desired on the treated paper.

Usually between 1 to 70, preferably from 25 2 to 50, and most preferably from 4 to 30, wt. % of paper treating agent is provided in the liquid treating composition.

The particular type of liquid vehicle is not critical so long as it performs the function of 30 assisting deposition of the paper treating agent onto the paper web. Illustrative liquid vehicles include water and organic solvents which are compatible with paper, and

preferably papermaking operations. Water is the preferred liquid vehicle.

The liquid composition used in the process of this invention will usually contain a foaming agent in an amount effective to provide a foam having the requisite structure. In some instances the paper treating agent may possess sufficient foaming properties to provide the requisite foam structure. In such cases the paper treating agent is also the foaming agent so that the presence of added foaming agent is not essential. The particular type of foaming agent is not critical but may be selected from foaming agents recognized by those skilled in the art as capable of forming the requisite foam. Typically, foaming agents are surfactants, i.e. surface active agents, which will operate to provide the requisite foam characteristics. Illustrative foaming agents include (1) nonionic or anionic surfactants, such as: ethylene oxide adducts of long-chain alcohols or long-chain alkyl phenols, such as mixed  $C_{11}$ - $C_{15}$  linear secondary alcohols containing from 10 to 50, preferably from 12 to 20, ethyleneoxy units,  $C_{10}$ - $C_{16}$  linear primary alcohols containing from 10 to 50, preferably from 12 to 20, ethyleneoxy units,  $C_8$ - $C_{12}$  alkyl phenols containing from 10 to 50, preferably from 12 to 20, ethyleneoxy units; fatty acid alkanolamides, such as coconut fatty acid monoethanolamide; sulfosuccinate ester salts, such as disodium N-octadecylsulfosuccinate, tetrasodium

N-(1,2-dicarboxyethyl)-N-octadecylsulfosuccinate, diethyl ester of sodium sulfosuccinate acid, dihexyl ester of sodium sulfosuccinic acid, dioctyl ester of sodium sulfosuccinic acid, or (2) cationic or amphoteric surfactants, such as: distearyl pyridinium chloride; N-coco-beta-aminopropionic acid (the N-tallow or N-lauryl derivatives) or the sodium salts thereof; stearyl dimethyl benzyl ammonium chloride; the betaines or tertiary alkylamines quaternized with benzene sulfonic acid. Such foaming agents are well known and any similar surfactant can be used in addition to those previously identified. Blends of more than one foaming agent may be used. In selecting the foaming agent for a particular foam, care must be exercised to use those agents which will not unduly react with the other agents present or interfere with the foaming or treating process.

The concentration of foaming agent in the composition is not critical but may be any amount sufficient to provide the requisite foam structure. The amount of foaming agent will vary depending upon the particular foaming agent, particularly paper

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treating agent, foam structure, rate of foam application, rate of moving paper, and the like considerations which vary among applications.

Typically, the amount of foaming agent is between

5                   0.1 to       5, preferably       0.5 to  
3, and most preferably       1.0 to       2.0,  
weight percent of the liquid treating composition.

10                  Additional adjuvants may optionally be provided to the fluid treating composition consistent with those procedures established in the art, including wetting agents, heat sensitizers; setting agents; dispersants; stabilizers; screening agents, antioxidants; foam stabilizers such as hydroxyethyl cellulose or hydrolyzed guar gum;

15                  to the extent they do not unduly affect the desired foam properties in application of the foam to the paper web. The concentration of adjuvants which may be provided follows those practices established in the art.

20                  The foam used in the process of this invention contains gas and the liquid treating composition. The gas is required as the vapor component of the foam. The gas may be any gaseous material capable of forming a foam with the liquid vehicle containing paper treating agent. Typical gas materials include air, nitrogen, oxygen, and inert gases. Air is the preferred gas. The relative proportion of liquid treating composition to gas is not critical beyond that amount effective to provide the required uniform foam structure.

25                  The relative proportion of liquid treating composition to gas is the amount sufficient to provide a foam having the required structure in

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terms of density, bubble size and half-life which provides fast-breaking, fast-wetting foam stability. The density of the foam is between 0.005 to 0.6, preferably from 0.01 to 5 0.4, g/cm<sup>3</sup>. The foams have an average bubble size of between 0.05 to about 0.5, preferably from 0.08 to 0.45, millimeters in diameter. The foam half-life is between 1 to 60, preferably from 10 3 to 40, minutes.

Foam density and foam half-life are determined by placing a specified volume of foam in a laboratory graduated cylinder of known weight, such as a 100 cm<sup>3</sup> or 1,000 cm<sup>3</sup> cylinder, determining 15 the weight of the foam in the cylinder, and calculating the density from the known volume and weight of the foam in the cylinder. From the measured foam density and volume, and the known density of the precursor liquid, the liquid volume 20 which would equal one-half of the total weight of the foam in the cylinder can be calculated. The foam half-life is the amount of time for this volume to collect in the bottom of the cylinder.

The foam bubble size can be determined by 25 counting the number of bubbles and measuring their diameters in a foam sample and calculating the average bubble diameter size from such measurements.

Foams which may be used in the process of this invention include those described in U.S.

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The particular sequence of addition of components in the treating composition is not critical, but may be achieved by mixing a liquid vehicle, paper treating agent, foaming agent, and other optional additives in any desired sequence, following those practices in the art.

10

The foam is produced using commercially available foaming apparatus, generally consisting of a mechanical agitator capable of mixing metered quantities of gas and liquid composition. The foaming step is controlled by adjusting the volume of gas introduced into the foaming apparatus and the rotation rate of the rotor in the foaming apparatus. The rotation rate is significant in providing a foam that will have the required bubble size and half-life. The relative feed rates of the liquid composition and gas will determine the density of the foam.

20

25

Once produced, the foam passes to a foam applicator. The foam fills an orifice between upstream and downstream lips of the foam applicator before contacting the paper web. The edges of the upstream and downstream foam applicator lips in contact with the paper web may be of any selected configuration, such as pointed, tapered, flat, beveled, arched or otherwise.

The foam applicator generally has sufficient side-to-side width that the foam can be applied across the entire width of the paper web.

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During operation, a pressure above ambient pressure develops in the application chamber of the foam applicator. This pressure is dependent upon several factors including foam density, the rate of foam passage, the rate of paper web passage, as well as the absorbency and porosity of the paper web. The pressure is maintained within the foam applicator to enable sufficient foam pressure against the paper web. This pressure, i.e. greater than zero, will generally range from 0.69 mbar to 0.69 bar (0.01 to 10), preferably from 6.9 to 207 mbar (0.1 to 3), and most preferably from 20.7 to 69 mbar (0.3 to 1 psi).

The lips of the foam applicator are sealed to provide a closed system and prevent loss of foam due to the pressure in the application chamber of the foam applicator. The seals between the lips of the foam applicator may be fixed or moveable to match variations in paper width.

Preferred foam applicators which may be used in this invention are described in U.S.

-A- 4,023,526.

The wet paper web passes across the lips of the foam applicator passing from the upstream lip to the downstream lip. Upon contact with the paper web, the fast-breaking, fast-wetting character of the foam provides immediate breaking of the components thereof and the liquid composition is rapidly absorbed into the wet paper web providing a uniform distribution and penetration.

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The rate at which the paper web passes across the foam applicator may vary over a wide range, covering those ranges typical in papermaking operations. Typically, the paper web will be supplied at a rate of at least about 60.8 5 200, preferably from (400 to 121.6 to 1824 6000), and most preferably from (500 to 152 to 1 064 m/min 3500, feet per minute).

The temperature at which the foam is produced and applied in the process of this 10 invention is not critical but may range from ambient up to 100°C or more in cases where the paper treating agent is heated prior to and/or during application to the paper web.

Single or multiple foam application steps 15 may be provided by the process of this invention. Foam may be applied to either or both sides of the paper web. In multiple or two-sided applications, the foam applicators may be supplied with one or more foamed treating compositions produced in one or 20 more foam generation means. In multiple or two-sided applications the amount and composition of the applied foam may be equal or different among the various applications. Multiple foam application steps may be in direct succession or separated by 25 other process steps, as may be used in typical papermaking operations.

The substrate passing across the foam applicator may be assisted by appropriate guide means to form the requisite contact along the 30 applicator lips. Guide means may be provided either upstream, downstream, or both, of the foam applicator. Typical guide means include paper

rolls, nips, bars, or similar devices effective in assisting the substrate to contact the lips across the entire width of the substrate. A preferred guide means is a vacuum powered holding device, preferably immediately preceding the upstream lip. described in cofiled U.S. Patent Application Serial No. 715,170 (Brown et al.), entitled "Vacuum Guide Used in Flexible Sheet Material Treatment", incorporated herein by reference.

The treated wet paper web is then dried using drying procedures established in papermaking operations. The moisture content of the dried paper will generally be reduced to less than 10, and most preferably from 0 to 5, wt. %.

The dried, treated paper may then be recovered using those procedures established in papermaking operations.

The processes of this invention are preferably applied to continuous paper treating operations, typical in papermaking and treating operations.

In a typical embodiment a wet paper web, as produced during a wet-laid papermaking operation, is conveyed across the foam applicator. A metered quantity of liquid treating composition containing paper treating agent, foaming agent and liquid vehicle, is foamed with a metered quantity of gas in a commercially available foaming apparatus, to produce foam having the requisite foam characteristics. The foam is passed through the foam applicator filling the orifice between upstream and downstream lips of the applicator, coming into contact with the paper web to provide uniform distribution of liquid treating composition onto the paper web. The treated, paper web is then dried and recovered using commercial available driers and paper rolls.

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The treated, paper web produced by the process of this invention has a substantially uniform distribution of paper treating agent while obtaining the desired treated properties, such as 5 dry and wet strength, after the treating process. It was unexpected that substantially non-porous, wet paper, such as wet-laid paper web produced during papermaking operations, could be effectively treated at high speeds, providing a substantially uniform 10 distribution of paper treating agent onto the wet paper by using the process of this invention.

The following examples are illustrative of some embodiments of this invention.

15

#### EXAMPLES

In these examples, paper web is treated under conditions designed to simulate wet-end paper web treatment during papermaking operations. The amount of foam applied to the paper web can be 20 calculated from the values given in the examples using the equation:

$$V_f = \frac{c_s \cdot v_s \cdot w_s \cdot \lambda}{c_l \cdot \rho_f} \quad (II)$$

wherein:

25  $V_f$  is the volume flow rate of foam, in  $m^3/min$  ( $ft.^3/min$ );

$c_s$  is the concentration of solids added to paper web, in wt. %;

$v_s$  is the line speed of the paper web, in  $m/min$  ( $ft./min$ );

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$w_s$  is the paper weight, in  $\text{g}/\text{m}^2$  ( $\text{lb.}/\text{ft.}^2$ );  
 $\lambda$  is the width across the paper web,

i.e., nozzle orifice, in m (ft);

5  $c_1$  is the concentration of solids in the  
foam composition, in wt. % of the pretreated web;

$\rho_f$  is the density of the foam, in  $\text{g}/\text{m}^3$   
( $\text{lb.}/\text{ft.}^3$ ) (equal to  $0.016 \text{ cm}/\text{cm}^3$ ).

10 Dimensional orientation as used in the  
context of this invention, unless otherwise  
indicated, is such that length is measured along the  
direction of paper movement and across the foam  
applicator lips, width is measured across the paper  
web and along the foam applicator lips and height is  
measured in terms of the direction perpendicular to  
15 the paper sheet.

20 Unless otherwise indicated, the following  
general procedure was used in the examples. Liquid  
treating composition was prepared by mixing, in the  
designated proportions, the designated components  
including the paper treating agent or agents,  
foaming agent and water. This composition was fed  
at the designated volume flow rate, along with air  
at the designated air feed rate, to a commercially  
25 available foaming apparatus, Model No. 4MHA Oakes  
Mixer, where the foam was generated, which foam was  
conveyed to the foam applicator.

30 The foam applicator consisted of an  
application chamber and a nozzle, having a width  
about equal to the paper width. Foam entered the  
application chamber from the foam generation means  
through a conduit  $9.5 \text{ mm}$  in diameter. The  
application chamber was about  $229 \text{ mm}$  high and had

an exit slot to the nozzle measuring 457 mm (18 inches) wide by approximately 3.2 mm (0.125 inches) long, i.e., as measured in the direction of paper movement. The nozzle was of similar width and length measuring 5 38 mm (1.5 inches) high. The nozzle had flat lips containing the paper web for 3.2 to 6.4 mm (0.125 to 0.25 inches) in length. Foam passed from the application chamber filling the orifice between the upstream and downstream lips to the designated foam 10 pressure. Paper web, having the designated moisture content, was fed across the nozzle, from the upstream to downstream lips, at the designated paper speed. The treated, paper web was then collected and dried on frames at 115°C (240°F) for 5 minutes in a 15 Despatch\* oven. The dried, treated paper was then recovered for testing and evaluation.

The treated, paper web was tested using well-established procedures as follows:

	Test	Procedure Used
20	Basis weight g/m <sup>2</sup> (lb./ream)	TAPPI Test Method T-410
	Cal., mm (mils)	TAPPI Test Method T-411
	Porosity, m <sup>3</sup> /m <sup>2</sup> ·min (ft <sup>3</sup> /min per ft <sup>2</sup> )	ASTM D 737
25	Dry tensile strength, kg/m (lb./in.)	TAPPI Test Method T-404
	Flex	TAPPI Test Method T-511
	Gurley stiffness, g.-cm.	TAPPI Test Method T-489
	Mullen, kg/cm <sup>2</sup> (lb./in. <sup>2</sup> )	TAPPI Test Method T-403

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Resins content, wt. %	TAPPI Test Method T-493
Stretch, %	TAPPI Test Method T-457
Wet tensile strength, kg/m (lb./in.)	TAPPI Test Method T-456

5        The designations used in the examples have  
the following meaning:

	<u>Designation</u>	<u>Description</u>
	CD	Cross direction
10	Foaming agent I	9 mole ethylene oxide adduct of mixed C <sub>11</sub> -15 linear secondary alcohols.
	Foaming agent II	7 mole ethylene oxide adduct of mixed C <sub>11</sub> -15 linear secondary alcohols.
15	Foaming Agent III	An ethyleneoxide adduct of linear alcohol distributed under the tradename Emersist®5953 by Emery Industries, Inc.
	Foaming agent IV	Sodium lauryl sulfate
20	Foaming agent V	Dodecyl benzene sulfonic acid.
	MD	Machine direction
	Tracer I	Green pigment distributed under the tradename Graphtol® by Sandoz Colors and Chemicals Co.
25	Tracer II	Red dyestuff distributed under the tradename Maxilon® Red GRL dyestuff by Ciba Geigy Inc.
	Tracer III	Fluorescent dye distributed under the tradename Leucophor AC by Sandoz Colors and Chemicals Co.
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Treating agent I      A polyamide wet strength agent.

Treating agent II      An aqueous phenolic resin dispersion containing 45 wt. % resin, used as a binder, distributed under the tradename BKUA 2370 by Union Carbide Corp.

5

Treating agent III      A melamine formaldehyde resin used as a crosslinker of paper and binder, distributed under the tradename Cymel®373 by an American Cyanamid Co.

10

Treating agent IV      An acrylic latex containing 45 wt. % resin, used as a binder, distributed under the tradename, UCAR® 879 Latex by Union Carbide Corp.

15

Treating agent V      Oxidized starch distributed under the tradename Stayco®M by A. E. Staley Manufacturing Co.

20      WPU      Wet pick-up defined as the weight percent of composition added to the paper web, based on the dry paper weight.

25      Examples 1 and Control A-F  
In these examples, wet-laid paper sheet of wood fiber having a density of 146.5 g/m<sup>2</sup> (4.32 oz/yd<sup>2</sup>) i.e., around 90 lb/ream, was treated using the previously described procedure. The liquid compositions used were as follows:

<u>Ingredients</u>	<u>Proportions wt. %</u>			
	<u>Control A</u>	<u>Control B</u>	<u>Controls C,E and Example 1</u>	<u>Controls D and F</u>
5 Treating agent I	-	-	31.25 <sup>a</sup>	20.83 <sup>b</sup>
Foaming agent I	-	0.5	0.5	0.5
Tracer II	-	0.2	-	-
Water	-	99.3	68.25	79.17

10 <sup>a</sup> - providing 2.5 wt. % active agent  
<sup>b</sup> - providing 1.7 wt. % active agent

15 In Example 1 dry paper was initially pre-watered to a moisture content of around 37.5 wt. %, i.e., 60 wt. % wet pick-up, followed by enclosure in a plastic bag for 24 hours to provide a uniform distribution of moisture content within the paper web, to simulate wet-end paper during papermaking operations. The pre-wetted paper web was then treated with the designated composition.

20 In Examples Controls B-F dry paper web was treated under various conditions. The process conditions for these examples as set forth in Table A as follows:

TABLE A  
EXAMPLES 1, B-F PROCESS CONDITIONS

	Pre-wetting	Treating Step	Example					
			1	B	C	D	E	F
Liquid feed rate g/min	350	122.5	183.3	183.3	122.5	367	245	
Air feed rate l/min	3.0	2.9	4.8	6.1	5.1	5.9	5.1	
Paper speed m/min	10	10	10	10	10	10	10	10
(ft/min)	(30)	(30)	(30)	(30)	(30)	(30)	(30)	(30)
Paper weight g/m <sup>2</sup>	146.5	146.5	146.5	146.5	146.5	146.5	146.5	146.5
(lb/ft <sup>2</sup> )	(0.03)	(0.03)	(0.03)	(0.03)	(0.03)	(0.03)	(0.03)	(0.03)
Paper width m	0.45	0.45	0.45	0.45	0.45	0.45	0.45	0.45
(ft)	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Total solids wt %	0	2.5	0	1.7	2.5	1.7	2.5	
Wet pick-up wt %	60	40 <sup>a</sup>	60 <sup>a</sup>	60 <sup>a</sup>	40 <sup>a</sup>	60	40	
Solids pick-up wt %	0	1.0	0	1.0	1.0	1.0	1.0	1.0
Foam density cm <sup>3</sup>	0.064	0.024	0.038	0.030	0.024	0.062	0.048	
Foam pressure bar	0.0345	0.0297	0.0172	0.0248	0.0248	0.031	0.0248	
(psi)	(0.5)	(0.43)	(0.25)	(0.36)	(0.36)	(0.45)	(0.36)	
bar		0.0152	0.0248	0.0248	0.0248			
(psi)		(0.22) <sup>a</sup>	(0.36) <sup>a</sup>	(0.36) <sup>a</sup>	(0.36) <sup>a</sup>			
Foamer kg/cm <sup>2</sup>	1.97	3.80	2.11	2.81	3.51		2.81	
(psig)	(28)	(54)	(30)	(40)	(50)	(- <sup>b</sup> )	(40)	

a - half on each side in two steps

b - not recorded

The treated, paper web, as well as untreated paper web designated Example Control A, were analyzed, using the test procedures described previously, with the results set forth in Table B.

5 Although the absolute values may vary among particular treating agents, the results demonstrate that treated paper properties, such as dry and wet tensile strengths, are provided by the process of this invention for treating wet paper web comparable  
10 to those properties provided by treating dry paper web under similar conditions.

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TABLE B  
Examples 1, A-F Paper Analysis

5	Example	Paper Treatment Conditions	Estimated Surfactant %	Resin Content %	Dry Tensile Strength		Wet Tensile Strength	
					kg/m (lb/in)		kg/m (lb/in)	
					MD	CD	MD	CD
	A	Untreated	0	0	503.7 (28.2)	276.8 (15.5)	25.0 (1.4)	14.3 (0.8)
	B	Surfactant only	0.3	0	385.8 (21.6)	207.2 (11.6)	23.2 (1.3)	10.7 (0.6)
10	C	30% WPU each side	0.3	1.16	534.0 (29.9)	307.2 (17.2)	150.0 (8.4)	91.1 (5.1)
	D	20% WPU each side	0.2	1.17	559.0 (31.3)	328.6 (18.4)	153.6 (8.6)	107.2 (6.0)
	E	60% WPU one side	0.2	0.94	498.3 (27.9)	296.5 (16.6)	112.5 (6.3)	67.9 (3.8)
15	F	40% WPU one side	0.2	1.24	548.3 (30.7)	305.4 (17.1)	116.1 (6.5)	80.4 (4.5)
	I	60% WPU one-side prewetted; 40% WPU treatment	0.5	1.05	476.9 (26.7)	266.1 (14.9)	142.9 (8.0)	78.6 (4.4)
20								

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Example 2

In this example, a binder was applied to wet-laid paper sheet of wood fiber, using the previously described procedures except that two nozzles were set one to each side of the paper with the foamed composition divided evenly to each nozzle. The paper was pre-wetted to a moisture content of 30% using a liquid composition of 0.5 wt. % Foaming agent I providing a foam density of 0.080 g/cm<sup>3</sup>. The treating composition contains the following ingredients:

	<u>Ingredients</u>	<u>Proportions</u>	<u>wt. %</u>
	Treating agent IV	88.888	
	Foaming agent I	0.25	
15	Tracer I	0.175	
	Water	10.937	

The process conditions used were as follows:

	Liquid feed rate g/min	90	
	Air feed rate l/min	1.6	
20	Paper speed m/min (ft/min)	10	(30)
	Paper weight g/m <sup>2</sup> (lb/ft <sup>2</sup> )	126.9	( 0.026)
	Paper width m (ft)	0.19	( 0.625)
	Total solids wt %	40	
	Wet pick-up wt %	40	
25	Solids pick-up wt %	16	
	Foam density g/cm <sup>3</sup>	0.055	
	Foam pressure mbar (psi)	7.59	(0.11,0.11 <sup>a</sup> )

<sup>a</sup> - upper and lower nozzle, respectively

Example 3

In this example, a composition of binder combined with crosslinker was applied to wet-laid paper web of wood fiber, using the procedures as described in Example 2. The paper was pre-wetted to a moisture content of 30% using a liquid composition of 0.5 wt. % Foaming agent I using a foam density of 0.08 g/cm<sup>3</sup>. The treating composition contained the following ingredients:

10	<u>Ingredients</u>	<u>Proportions wt. %</u>
	Treating agent IV	59.3
	Treating agent III	4.44
	Water	36.258
	Foaming agent IV	0.5

15 The process conditions used were as follows:

	Liquid feed rate g/min	143
	Air Feed rate l/min	1.7
	Paper speed m/min (ft/min)	10 (30)
	Paper weight g/m <sup>2</sup> (lb/ft <sup>2</sup> )	97.6 ( 0.02)
20	Paper width m (ft)	0.20 ( 0.65)
	Total solids wt %	27.795
	Wet pick-up wt %	80 <sup>a</sup>
	Solids pick-up wt %	22.236
	Foam density g/cm <sup>3</sup>	0.084
25	Foam pressure mbar (psi)	17.2 22.4 (0.25, 0.325 <sup>b</sup> )

<sup>a</sup> - half on each side

<sup>b</sup> - upper and lower nozzle, respectively

Example 4 and Controls G,H

In this example, a combination of binders and crosslinker were applied to wet paper sheet of wood fiber using the previously described procedures. The liquid compositions utilized were as follows:

Ingredients	Proportions/wt. %	
	Examples G&H <sup>a</sup>	Example 4
Tracer I	0.2	0.2
Treating agent IV	72.666	48.45
Treating agent II	9.4	6.03
Treating agent III	1.766	1.178
Water	16.17	44.342
Foaming agent IV	1.0	1.0

<sup>a</sup> - Composition of Example G diluted 50% in Example H.

In Examples Control G and H each side of the initially dry paper was treated. In Example 4 prewetted paper having a moisture content of 17% was treated. The treated, paper webs were dried for 3 minutes at 93°C followed by curing for 30 seconds at 149°C. The process conditions utilized were as follows:

	Example		
	G	H	4
Liquid feed rate g/min	98.3	147.4	147.4
Air Feed rate l/min	3.8	4.3	6.7
Paper speed m/min (ft/min)	10 (30)	10 (30)	10 (30)
Paper weight g/m <sup>2</sup> (lb/ft <sup>2</sup> )	117.2 (0.024)	117.2 (0.024)	117.2 (0.024)
Paper width m (ft)	0.45 (1.5)	0.45 (1.5)	0.45 (1.5)
Total solids wt %	37.72	25.04	25.04
Wet pick-up wt %	40	60	60
Solids pick-up wt %	15.1	15.1	15.1
Foam density g/cm <sup>3</sup>	0.026	0.034	0.022
Foam pressure mbar (psi)	12.4 11.0 (0.18) (0.16 <sup>a</sup> )	12.4 10.3 (0.18) (0.149 <sup>a</sup> )	24.8 (0.36)
Foamer kg/cm <sup>2</sup> (psig)	4.08 (58)	3.66 (52)	<sup>b</sup>

<sup>a</sup> - upper and lower nozzle, respectively  
<sup>b</sup> - not recorded

The treated, paper webs were analyzed, using the test procedures previously described, with the results set forth in Table C. The results demonstrate that essentially equivalent paper properties are obtained when applying treating agents to wet paper by the process of the invention as compared to treating dry paper.

5

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TABLE C  
Example 1 and Controls G-H Paper Analysis

		Example H	Example G	Example G	Example H	Example 4	Example 4
5	Post treatment heating	dried	dried	cured	cured	dried	cured
	Dry MD:	339 (19)	276 (15.45)	297 (16.65)	395 (22.1)	355 (19.9)	455 (25.5)
10	Tensile Strength kg/m CD: (1b/in)	220 (12.3)	185 (10.35)	206 (11.55)	264 (14.8)	223 (12.5)	286 (16)
	Dry MD:	54 (3)	51 (2.85)	106 (5.95)	123 (6.9)	70 (3.9)	89 (5)
15	Tensile Strength <sup>a</sup> kg/m CD: (1b/in)	41 (2.3)	30 (1.7)	80 (4.50)	96 (5.4)	46 (2.6)	95 (5.3)
	Mullen kg/cm <sup>2</sup> (psi)	2.86 (40.7)	2.42 (34.4)	2.37 (33.7)	3.00 (42.7)	2.77 (39.4)	3.05 (43.4)
	Stiffness MD: (g -cm)	2650 CD: 1428	2738 1456	2968 1372	2891 1525	2310 1589	2800 1540
20	Porosity m <sup>3</sup> /m <sup>2</sup> min (CFM/ft <sup>2</sup> )	23.3 (76.82)	25.3 (83.38)	24.3 (79.90)	23.0 (75.70)	24.2 (79.50)	23.6 (77.50)
	Basis						
	Weight g/m <sup>2</sup> (1b/ream)	139.4 (82.01)	141.2 (83.08)	143.1 (84.19)	137.5 (80.89)	136.9 (80.56)	142.3 (83.73)

<sup>a</sup>-after boiling

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Example 5 and Controls I-J

In these examples a combination of binders and crosslinker were applied to filter paper. The liquid compositions used were as follows:

Ingredients	Proportions/wt. %	
	Control J	Example 5
Treating agent IV	55.55	79.36
Treating agent II	11.10	15.873
Treating agent III	1.10	1.086
Tracer I	0.10	0.20
Foaming agent I	0.50	0.50
Water	32.15	3.48

For comparison, dry paper web was treated in Example Control J whereas in Example 5 the filter paper was prewetted to a moisture content of 17.6 wt.% using a 0.5 wt. % composition of Foaming agent I providing a foam density of 0.04 g/cc. The process conditions used were as follows:

	Control J	Example 5
Liquid feed rate g/min	149	104
Air feed rate l/min	3.4	3.0
Paper speed m/min (ft/min)	4.56 (15)	4.56 (15)
Paper weight g/m <sup>2</sup> (lb/ft <sup>2</sup> )	117.2 (0.024)	117.2 (0.024)
Paper width m (ft)	0.45 (1.5)	0.45 (1.5)
Total solids wt %	30.6	43.9
Wet pick-up wt %	60	42
Solids pick-up wt %	18.4	18.5
Foam density g/cm <sup>3</sup>	0.044	0.035
Foam pressure mbar (psi)	7.6	7.6
Foamer kg/cm <sup>2</sup> (psig)	(0.11)	(0.11)
	(0.18 <sup>a</sup> )	(0.18 <sup>a</sup> )

a- upper and lower nozzle, respectively

The properties of the treated paper as well as untreated paper designated as Control I, were

tested using the previously described procedures, with the results set forth in Table D as follows:

TABLE D

Examples 5 and Controls I-J Paper Analysis

<u>Test</u>	<u>Control I</u>	<u>Control J</u>	<u>Example 5</u>
Resin Content %	0	16.9	14.5
Cal mm (mils)	0.49 (19.2)	0.53 (21.0)	0.52 (20.7)
Dry tensile strength kg/m (lb/in)	75.0 (4.2)	441.1 (24.7)	394.7 (22.1)
Dry tensile strength <sup>a</sup> kg/m (lb/in)	17.9 (1.0)	210.8 (11.8)	207.2 (11.6)
Gurley stiffness g·cm	693	3276	2310
Flex	5.2	1000+	1000+
Mullen kg/cm <sup>2</sup> (psi)	0.4 (5.6)	3.0 (42.5)	2.6 (36.7)
Porosity m <sup>3</sup> /m <sup>2</sup> ·min (CFM/ft <sup>2</sup> )	27.8 (91.4)	23.5 (77.3)	22.3 (73.4)
Stretch %	0.9	3.6	4.2
Stretch <sup>a</sup> %	4.0	7.1	8.0

<sup>a</sup> - after boiling

The results demonstrate that the application of paper treating agents by the process of this invention provides comparable product performance as compared with dry-end paper treatment under similar conditions.

Example 6

In this example, internally sized vellum paper weighing about 89.6 g/m<sup>2</sup>, having a moisture content of from 5.2 to 5.7 wt. %, was pre-treated with water and plastic wrapped for several days to provide a wetted paper having a moisture content of 19%.

A starch-treating composition was prepared by cooking 30 wt. % Treating agent V in water for about 30 minutes at about 90°C. The cooked composition was diluted to 20 wt. % starch and held thereafter at about 75°C. Sufficient Foaming Agent V was added to the composition to provide about 0.12 wt. % foaming agent. The composition, containing 0.25 wt. % added Tracer III, was foamed using an 8-inch radial type foam generator, with liquid metering pump, metered compressed air supply, and a foam applicator consisting of a foam distribution chamber, a foam application chamber fitted with a pressure gauge and foam applicator nozzle lips suitably jacketed to enable maintenance of foam temperature at about 66°C. The foamed composition was applied under the following conditions:

TABLE IX

	Paper moisture content °C	19
	Paper sheet speed m/min (ft/min)	500 (1500)
20	Treating agent wt. %	12
	Foaming agent wt. %	V (0.25)
	Wet coat weight g/m <sup>2</sup>	10.8
	Dry coat weight g/m <sup>2</sup>	1.3
25	Foam pressure mbar (psi)	40 (0.58)
	Foam density g/cm <sup>3</sup>	0.28
	Foam temperature °C	66
	Paper temperature	Ambient
	Coating uniformity	Good

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PATENT CLAIMS

1. A process for producing treated paper comprising:

(A) producing a wet paper web having a moisture content of at least about 5 weight percent;

(B) producing a foam containing gas and a liquid treating composition comprised of liquid vehicle, paper treating agent and foaming agent, which foam has a density of from 5 to 800 g/m<sup>3</sup>, an average bubble size of from 0.05 to 0.5 mm in diameter and a foam half-life of from 1 to 60 minutes;

(C) passing the foam through at least one foam applicator nozzle filling an orifice between upstream and downstream lips of the nozzle with the foam;

(D) passing the wet paper web across the nozzle in the direction of the upstream lip to the downstream lip;

(E) applying a controlled amount of the foam to the surface of the wet paper web to provide a uniform distribution of the paper treating agent onto the wet paper web;

(F) drying the wet paper web; and

(G) recovering the dried, treated paper web.

2. The process of claim 1 wherein the paper treating agent is a sizing aid, binder or coloring agent.

3. The process of claim 2 wherein the paper treating agent is starch, wet-strength or dry strength resin, dye or pigment.

4. The process of claims 1 to 3 wherein the wet paper has a moisture content of from at least about 5 wt. % up to the saturation level of the paper or of from 5 wt. % to 40 wt. %.

5. The process of claims 1 to 4 wherein the gas is air and the liquid vehicle is water.



EUROPEAN SEARCH REPORT

0195458  
Application number

EP 86 10 3923

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
X, Y	GB-A-1 585 874 (UNION CARBIDE) * Claims 1-5,7; page 2, line 22 - page 3, line 9 * & DE - A - 2 722 083 (Cat. D)  ---	1-3,5	D 21 D 3/00
D, Y	US-A-4 158 076 (H.I. WALLSTEN) * Whole document, in particular column 13, lines 54-56 *	1-5	
Y	---	1-5	
Y	TAPPI, vol. 51, no. 4, April 1968, pages 83A-86A, Atlanta, GA, US; W.P. HAMILL: "Coating paper with foam" * Pages 84A,85A *	1-5	
Y	---	1-5	TECHNICAL FIELDS SEARCHED (Int Cl 4)
A	GB-A-1 039 540 (REED PAPER GROUP) * Claims 1-21 *		B 05 C D 21 D D 21 H
A	---		
A	DE-B-1 221 093 (VEREINIGTE PAPIERWERKE SCHICKEDANZ)  ---		
A	H. HENTSCHEL: "Chemische Technologie der Zellstoff- und Papierherstellung", 1967, 3rd edition, pages 546-552, VEB Fachverlag, Leipzig, DD; "Trocknung und Fertigstellung des Papiers"  -----		
The present search report has been drawn up for all claims			
Place of search	Date of completion of the search	Examiner	
THE HAGUE	13-06-1986	NESTBY K.	
CATEGORY OF CITED DOCUMENTS			
X : particularly relevant if taken alone	T : theory or principle underlying the invention		
Y : particularly relevant if combined with another	E : earlier patent document, but published on, or after the filing date		
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